



PATENT

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Dated: January 14, 2005

BY:

Rodney D. DeKouif
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: David C. Dunand)
Serial No: 10/680,639)
Filed: October 7, 2003) Attorney Docket No. 6513-DIV
For: SUPERCONDUCTING)
Mg-MgB₂ AND RELATED)
METAL COMPOSITES)
AND METHODS OF)
PREPARATION)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

RULE 131 DECLARATION OF DAVID C. DUNAND

1. I, David C. Dunand, am inventor of the invention (the "Invention") disclosed and claimed in the above-entitled application (the "Application"). I am a Professor in the Department of Materials Science and Engineering at Northwestern University. I make this declaration in support of the Application and, in particular, to antedate a reference cited against the Application.

2. The Invention claimed in the Application was completed before the effective date of the journal article by Sharoni, et al., entitled "Spatial variations of the superconductor gap structure in MgB₂/Al composite," *J. Phys. Condens. Matter* 13 (2001) L503-L508 (*i.e.*, the Sharoni reference). More specifically, the

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JAN-13-2005 03:32P FROM:

Invention was conceived and with due diligence reduced to practice prior to the effective date of the Sharoni reference. (The effective date of publication, i.e., June 4, 2001, can be found at <http://www.iop.org/EJ/toc/0953-8984/13/22>.)

3. This Declaration, and prior invention, is supported by copies of pertinent pages from my laboratory research notebook, entries which were contemporaneously witnessed by Graduate Student Naomi Davis. Date redacted copies of the aforementioned notebook pages are provided collectively as Exhibit A and incorporated herein by reference. These documents establish that the Invention was made at least as early as March 26, 2001, which is a date earlier than the effective date of the Sharoni reference.

I hereby declare that: All statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; that those statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code; and that willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Date January 13, 2004

David C. Dunand
David C. Dunand

Monday,

I discussed last week with D.N. Seidman the idea of superconducting composites based on the new compound HgB_2 . I had mentioned to him the following simple idea:

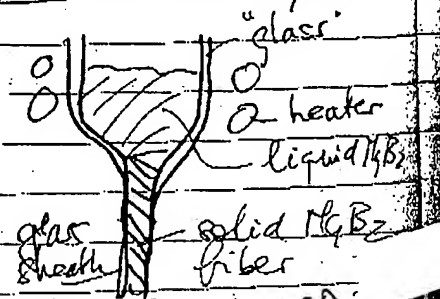
① Infiltrated $\text{Hg}-\text{HgB}_2$ composites

The $\text{Hg}-\text{B}$ phase diagram shows that Hg and HgB_2 are at equilibrium with each other. It should thus be possible to infiltrate a packed bed of HgB_2 powder (which may have been pre-sintered to make a continuous skeleton) with liquid Hg . The net result is a composite with a continuous superconducting phase of HgB_2 embedded in a continuous metallic Hg phase, which allows for thermal management (conducting heat away from HgB_2 and preventing loss of superconductivity) and also being able to carry current if s.c. is lost.

The following other ideas came to me, undisclosed to D.N. Seidman:

② Fabrication of HgB_2 fibers

Superconducting fibers would be very useful embedded in a non s.c. matrix (polymer, metal, glass, ceramic) which provides thermal and mechanical properties. Existing s.c. fibers can be made by drawing (when ductile) or by powder sintering (when brittle). My idea uses a different technique, i.e. the Taylor wire technique. HgB_2 powder is packed in a "glass" tube (any amorphous ceramic such as Pyrex, E glass, etc). The HgB_2 is melted and the glass is quickly drawn into a hollow fiber containing HgB_2 which solidifies into a continuous fiber, which can be spooled.



4/20/77 J. Van

Read & understood 1/9/80

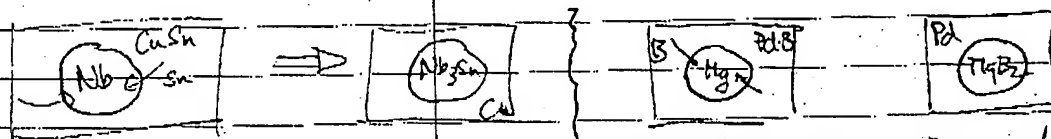
Taylor wires have been made with many metals (see Donald, 1987).

The following requirements are needed: (1) no reaction between glass and MgB_2 , (2) working temperature of glass must be higher than MgB_2 , (3) glass must become highly viscous before MgB_2 solidifies, lest it deforms and breaks the MgB_2 . Pyrex seems a good first choice.

The glass sheathed MgB_2 fiber can then be embedded in a matrix (metal, glass, ceramic, polymer) to form a composite by usual composite processing techniques. Alternatively, the glass can be dissolved by an acid (HF-containing acid) and the bare MgB_2 fibers can be used in a composite. Some dissolution of MgB_2 may be tolerable as part of the glass removal.

③ Ductile $Mg-B-Pd$ wires as precursors for $Pd-MgB_2$ composites

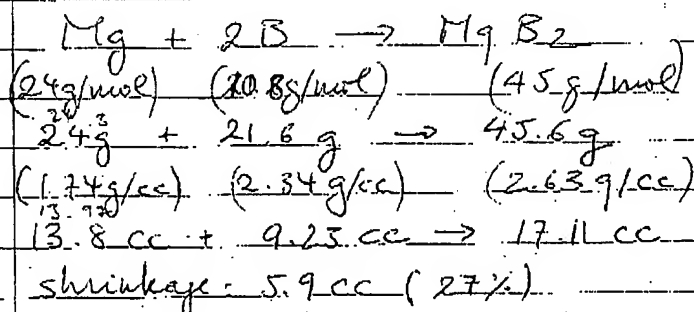
Nb_3Sn-Cu composites are ideal, as Cu prevents thermal runaway. They are produced by sticking Nb wires between a Cu-Sn bronze matrix and drawing, resulting in a ductile Cu-Sn/Nb composite which can be drawn and shaped (coiled, etc.) to shape. Heat-treatment diffuses Sn to the Nb fibers, forming the brittle Nb_3Sn superconductor in a Cu matrix.



The same idea can be used for MgB_2-X composites, where X is a ductile metal which can be alloyed with B . A good example is Pd , which can accept 2.2 wt% B (at%) So , Mg wires could be embedded within sheets of $Pd-B$ alloy, diffusion-bonded, drawn or shaped as needed. Upon heating below $650^\circ C$ (melting point of Mg),

calculations for volume fractions of Pd/MgB₂ composite

• MgB₂ synthesis



$$\frac{9.23}{13.8 + 9.23} = 0.398$$

$$\frac{9.23}{17.11} = 0.539$$

• Pd-B alloy Pd-2.2wt% B (max solubility)

$$21.6\text{g B} \rightarrow 960.2\text{g Pd}$$

• Mg-B-Pd alloy

$$960.2\text{g Pd} \quad (12\text{g/cc}) \rightarrow 80.0\text{cc Pd}$$

$$45.6\text{g MgB}_2 \rightarrow 17.11\text{cc MgB}_2$$

$$\begin{array}{|l}
 83.4\text{vol}\% \text{ Pd} \\
 17.6\text{vol}\% \text{ MgB}_2
 \end{array}$$

→ 1/6 of the volume is MgB₂

$$\frac{\pi a^2}{4b^2} = 0.176$$

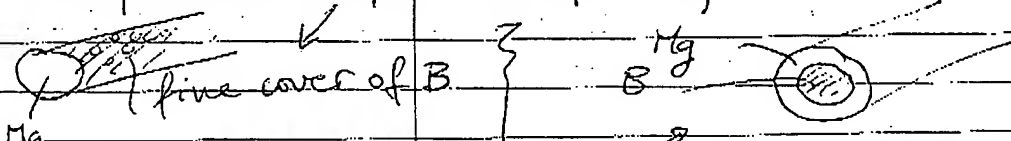
$$\frac{a}{b} = \sqrt{0.176 \frac{4}{\pi}}$$



This volume fraction is not very high, but still commercially feasible, provided price of Pd is not too high.

NB: some Mg will dissolve in Pd.

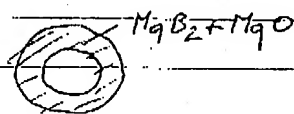
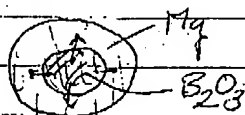
- Additional B could be introduced by dipping Mg wires in B suspension before compacting with Pd or Pd-B foils.



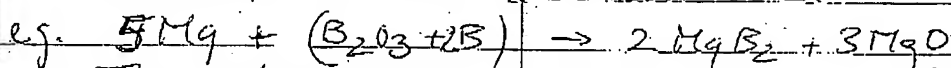
or filling Mg tubes with B powder and then compacting with Pd. Extrusion would still be easy with B powder.
read + understood *Marvin G. Lang*

- Alternative is to fill Mg tubes with B_2O_3 , which is viscous at extrusion temperatures. The reaction is then

$$4Mg + B_2O_3 \rightarrow MgB_2 + 3MgO$$



or a suspension of B particles in B_2O_3 , which will remain viscous & drawable.



The advantage is that the metallic matrix can now be a metal different from Pd, and the volume fraction of fibers has no restrictions.

MgO_2 fibers (160 μm in ϕ) have been disclosed at the APS meeting. B fiber is exposed to Mg vapors and forms MgB_2 which was found to be superconducting.

Taylor wire technique has following advantages:

- much more rapid production
- diameter easily changed (1-100 μm typically)
- different grain structure
- sheath of glass.

Good matrices for "filled tube" process: any metal which does not dissolve into Mg (which would contaminate MgB_2)

1. Cu: zero sol. in Mg; eutectic at 48°C; $MgCu_2$ & Mg_2Cu ; 22wt% Mg in Cu
2. Ni: zero sol. in Mg; $MgNi_2$ & $MgNi$; zero

read & understood,

1/20/91

Solubilities

Metals	$\% \text{ in Mg}$	Alloys	Intermetallics	Eutectic (C)
Cu	0	~3wt%	2	483
Ni	0	0	2	506
Co	0.12	~1.5at%	0	651
Fe	0	0	1	635
Cr	0	0	0	—
Au	0	huge	many	575
Be	0	0	1	—
Pd	0.23	5wt%	7	540
PE	0	0	5	575
Nb	0	0	0	—
Mo	0	0	0	—

Other advantages:

high conductivity: Cu (but reduced by Mg in solid soln)
 Au (but δ)

Processing: Cu low melting
 electrodeposition: Cu, Ni, Cr, Au, Pd, Pt

no bowdles Cu, Au
 read & understood
 Name of line

Other Idea:

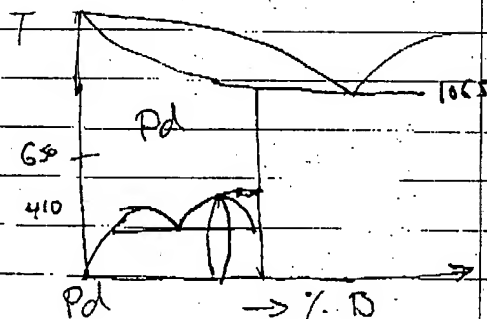
React B with Cu-Mg melt $2B + Cu + Mg \rightarrow MgB_2 + Cu$
 this is only possible if Cu does not dissolve and
 deteriorates MgB_2 s.c. properties

1. infiltrate B fibers or B skeleton
 with Cu-Mg melt
2. react in the melt
3. solidify Cu-MgB₂ composite.

This will work with non-boride formers, i.e. mostly Cu and Au

Read: 11/1/84

Thomson Jan



pure B dissolves Cu ($\sim 16 \text{ wt}\%$) at 1013°C

1 mol B = $4.61 \text{ cm}^3 \sim 40 \text{ vol}\%$

$\frac{1}{2}$ mol Mg = $6.89 \text{ cm}^3 \sim 60 \text{ vol}\%$

30 vol% B } $\sim 50 \text{ vol}\% MgB_2 \rightarrow \frac{2}{3} MgB_2$
 45 vol% Mg }
 25 vol% Cu 25 vol% $\frac{1}{3} Cu$

BMG → batch program works! → code
 some cryst. matrix! → new paper with double
 no creep error due to reloading after
 2 h hold → throw away data
 stand alone { - compression
 paper { - hold
 matrix info

• MRS paper → draft on Monday
 practice talk on Thursday

Tasker Carr Search Corn

↳

↳ carbon steel

Taylor wire MgB_2 trial

(Went to glass blowing shop and tied with pyrex (10 mm))

- can draw easily empty wire

- MgB_2 powder (~ 1 cm high) gets red hot, but no melting visible
 Can partially draw pyrex, but eventually fractures. MgB_2
 does not burn.

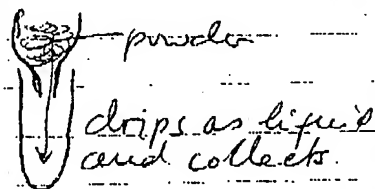
Problem is ϕ is too large, cannot keep the whole block
 of MgB_2 hot. → try pyrex tube, maybe try a torch
 with multiple outlets.

Tried finer wires $\sim 4 \mu\text{m}$, and after a few trials, got a wire $\sim 100-200 \mu\text{m}$, with black MgB_2 inside. It is not clear whether the powder was melted throughout, but it seemed to be

• tried Cu in $4 \mu\text{m}$ tube (pyrex), but not successful due to high T_m (glass leaked before Cu melted)

• Zn powders worked, nice Taylor wires.

• Tried again MgB_2 and got a nice length $\sim 10 \text{ cm}$ uninterrupted. Pyrex was not cleaned, so bubbles interrupt wire in some parts. Maybe also need to "pre-melt" materials, e.g. as done for AgCl during their (but $T_m = 455^\circ\text{C}$ vs 800°C for MgB_2)



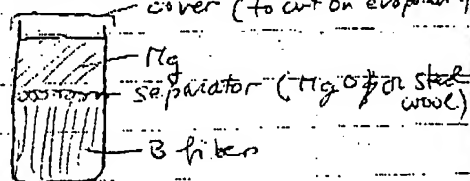
glass shop closed

read Canfield paper (PRL 86, 2423) and had the following

idea: integrated fiber formation & composite fabrication

1. Put B fibers and Mg into crucible

2. Heat at $950^\circ\text{C} \rightarrow \text{Mg}$ melts and seals crucible
hold for 2h $\rightarrow \text{Mg}$ vapor react with B fibers and form MgB_2 fibers like Canfield's paper



3. Pressurize with gas and force Mg into fiber preform
 $\rightarrow \text{MgB}_2 - \text{Mg}$ Composite

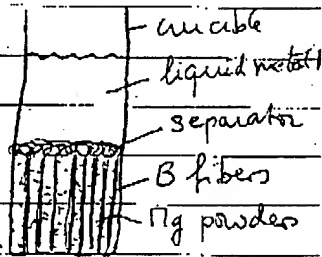
One of the main advantages is that MgB_2 fibers are formed in situ. They do not have to be handled, bundled, etc, which would easily damage or break them, because Canfield describes them

Another possibility is to infiltrate B-fibers with molten Mg and keep the composite at 950°C for 1-2 h, long enough for complete reaction to form MgB_2 . Solidifying gives a Mg composite with MgB_2 fibers.

Method to produce MgB_2 metal matrix composites

Campfield's paper shows that MgB_2 fibers can be produced by exposing B fibers to Mg vapors at 950°C for 2 h. However, the fibers are very brittle and bent after fabrication. It will be difficult to bundle them and subsequently infiltrate them without breakage. A solution is to synthesize and infiltrate the fibers in two closely consecutive steps, without handling the fibers.

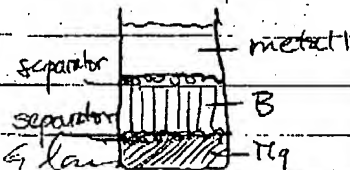
Step 1: Heat-up assembly to synthesis temperature. Mg melts and vaporizes, reacting with B-fibers to form MgB_2 . If the metal 1 is melted, it forms a liquid seal, thus preventing escape of Mg vapors. A non-wetting separator prevents metal 1 from contacting fibers. Examples for metal 1: Mg, Al, Cu alloy with low ~~melting~~ solidus (bronze, brass).



Step 2: pressurize with gas the crucible, forcing liquid metal 1 through the separator and between the MgB_2 fibers, thus making a composite. Solidify and extract composite from crucible. If metal 1 has higher melting point than synthesis temperature of fibers, first raise temperature to melt it.

Complex shapes could be made by this process.

A variation is to put Mg below fibers with separator permeable to Mg vapors. read + understood from 4 Jan



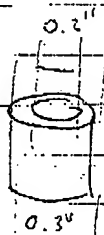
see p. 136: 24 g Mg
+ 21.6 g B

13.8 cc Mg 60 vol %
9.23 cc B ~ 40 vol %

so at best: 20 vol % B

30 vol % Mg powder

50 vol % porosity



$$\phi 5 \text{ mm} \quad A = 19.6 \text{ mm}^2 \rightarrow 3.9 \text{ mm}^2 \text{ B}$$

each fiber is 100 μm $\phi \rightarrow A = \pi \cdot 0.05^2 = \underbrace{0.2 \left(\frac{5}{0.1} \right)^2}_{500 \text{ fibers}}$

drop into 0.3" crucible -

@ 2 cm \rightarrow 10 m

Observe Dorian run infiltration

1. Turn H₂O on (circulate first at ~125 psi)
open to machine → flow switch on
zero pressure transducer
2. Close vessel - slide in
- put PB sheet
- tighten nut until lid is flush with vessel
3. Connect 1 p-transducer (showing as psix5, full vac = -65 to 0)
4. TC (check then)
5. Open gas tank, set 2nd stage regulator at 150 psi
5. purge 3 times - use valve 1 only
6. check for vac. leak - insulate vessel
verify vac. is constant for 45 min
7. Switch on main power, turn to 50% on both variacs
→ upper zone: 5.5 A / 100 V
→ lower zone: 4 A / 100 V
wait ~1½ h → 350°C
8. put T recorder on
turn to 75% (top only)
→ upper zone: 8.5 A / 150 V
wait ~45 min → turn to 90% (top only) (70% bottom)
wait ~40 min → turn down to 50% (both)
and fine-tune
- 45 min
9. Infiltrate
1. increase to 550 psi (regulator)
2. open valve #2 to vessel
3. switch off power
4. open valve #1 and pressurize to 550 psi (35 atm)
5. close valves 1 & 2 (will leak ~1-2 psi/min)
10. Cool down ~1 h to 200°C
turn off water, vent (use vent valve)
open

17g 99.99% 2 pieces from Emma (26.33g)

started cutting pieces

cut tubing & cleaned tubing

tubes: 0.555" ID = 14.10mm
0.305" ID = 7.75mm

filling in small tube to 2cm: $V = 943 \text{ mm}^3 = 0.943 \text{ cm}^3 \rightarrow$
assuming 50% packing: 0.472 cm^3

$0.472 \text{ cm}^3 \rightarrow 2.63 \text{ g/cc} \rightarrow 1.24 \text{ g MgB}_2$
1.70 $\rightarrow 0.802 \text{ g Mg}$ double (1.60 g Mg)

for large tube, multiply by $\left(\frac{0.555}{0.305}\right)^2 = 3.3 \rightarrow 4.1 \text{ g MgB}_2$
5.3g Mg

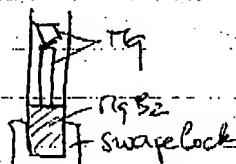
called by ()
else will send ~ 30 feet of 4 mil B fibers for free
Goodfellow charges a \$100 for 10m. [minimum order: 1.16 \$792 for 4 mil
5mm W core, 100µm diameter, 20.2 mg/m] \$1159 for 6 mil
 $\rightarrow 10 \text{ m} = 0.202 \text{ g}$

prepare small crucible: 17g: (1.605 g) (2 pieces)

add MgB_2 to crucible bottom for: 47.210g

-62g top + push with rod
48.208
383
48.441g

add pieces of Mg on top



$\rightarrow 1.231 \text{ g MgB}_2$

measured height: 6.5mm²²
not right, was not "touching
bottom" at first!

4.5mm for 0.233g \rightarrow ~ 22.5mm for full mass, OK!

leave for evening

stop

146
Sue Abkowitz

	RT E (ksi)
Ti64	: 16.88
Ti64+10W	: 15.67
	↳ (5.60g)

600°F
G (ksi)

modules measurement

5.041	→ 5.052	→ 5.056	→ 5.060	→ 5.067
0	5 min	10	15	20 min

-10%

0.02

change 0.4% in 20 min

• No dissolution: no, T too low, G increases

• W burn off: no, " "

(check if Ar is used)

• $\beta \rightarrow \alpha$ transf. is most likely!

mass: 4.633
volume $\frac{14.1}{1.5} = 2.35 \text{ cm}^3$
↳ 1.98 g/cm³

Alfa Aesar

prepare large crucible

4.633 g MgB₂ (15 mm height)

5.768 g Mg - 29.9% (2 large pieces, 1 small)

- put a thin 200 felt layer on top of Mg, stuff crucible to the top w. 200 felt (to prevent powder blow out during vac)
- load sample in vessel, contained in 503 crucible/pot
- evacuate/purge 3 times

11:35 vacuum -76, insulate vessel (leak check)

12:15

up low -75, 20 K, no leaks

12:18

50% 50% T₁ T₂ T₃ T₄ Vac

12:20

60% 60% 191 -73

1:40

" " 516 -70

2:00

75% 75% 582 568 -70

2:35

65% 65% 742 743 743 (734) -69

2:43

133 134 134 735

2:49

68% 68% 732

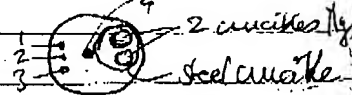
2:55

" " 734 734 739 734 switch off power

2 minute resuscitation to 2400 (980 psi)

3:40 depressurize 170°C, 1400 (200 psi)

Mg is at level of TC4



new large crucible old Hg powder from previous exp. 3.475g
new 0.939g

old Hg 4.558g 2 pieces
new Hg 2.4704 1 piece

4.417g
(Alfa Aesar)

7.028g (99.99%)

add $0.30 \pm 0.1g$ Hg to small crucible

both crucibles leave Hg to the rim, put no felt on top

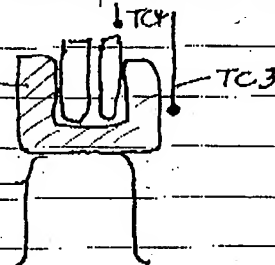
• load crucible in infiltrator

put them in thick graphite crucible
on top of s.s. crucible

much less insulation as last time
should get better heating

TC3 is next to gr. crucible

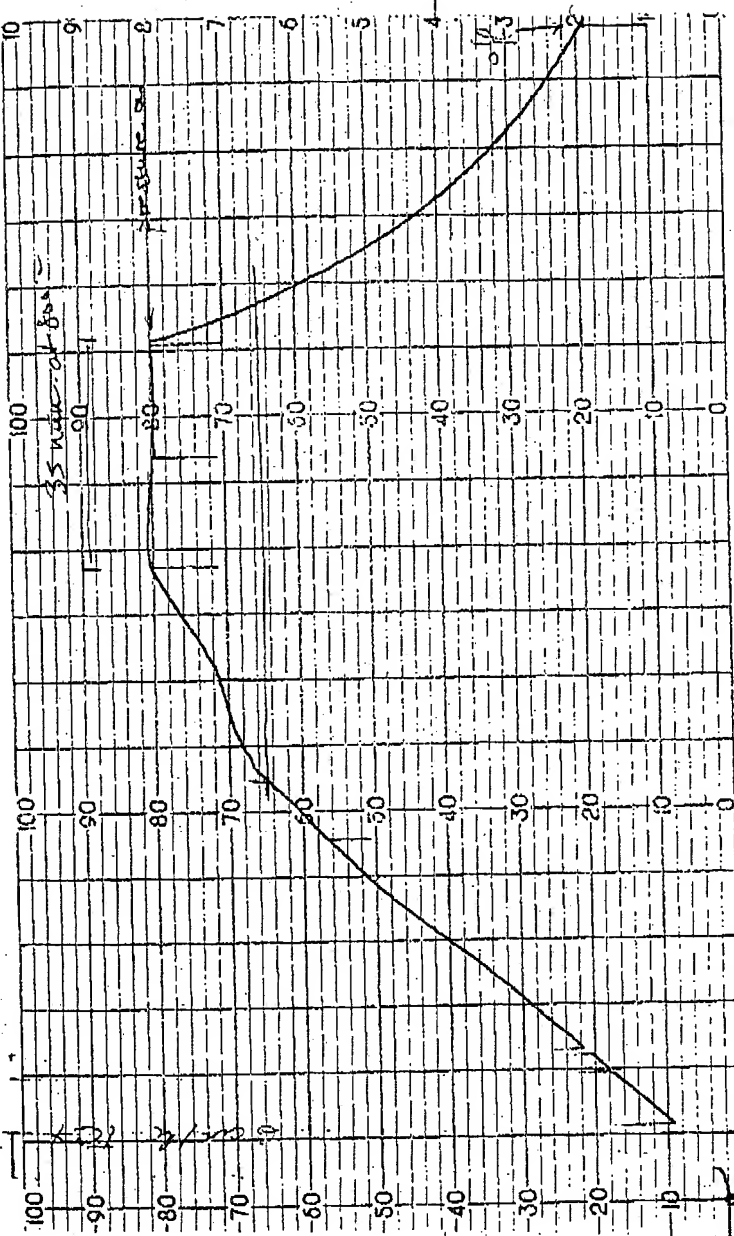
TC4 is just above steel crucibles



• evacuate to -67, insulate & leave for the night 11:35 PM

9³⁰ AM, vac at -41, good cal¹ Evacuate & flush twice

	P	T ₁	T ₂	T ₃	T ₄	upper lower	upper lower	
9:55	-55	24	22	19	21	70%	70%	change Ar bottle
10:25	-55	406	382	370	295	"	80%	
10:47	-53	568	569	590	538	75%	85%	
11:14	-51	688	702	732	712	80	95%	
11:30	-50	797	804	825	801	80%	95%	
11:45	-49	795	795	801	799	"	80%	
12:05	-49				803	"	"	switch off infiltrate
12:07	2525				755	0	0	in Goseco
12:10	2232				662			
12:39	1468				280			
12:52	1312				195			pump out, open



35 inches of bar

Pressure

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CHART NO. 820 195-6970-00

Carbon crucible contains 4.0 g of ~~dark~~ coarse, grey drose
could be Mg evaporated and condensed?

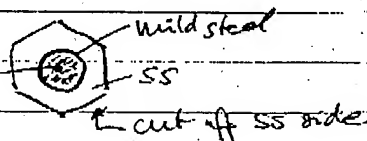
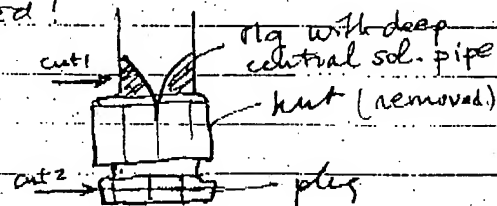
▲ cut large crucible → Mg has melted!

small oxide "hull" left in upper part,
(very light) ^{0.43g} Some white deposit on
top (MgO?). Cut

Cut 1: hack saw, see pipe hole

Cut 2: diamond saw.

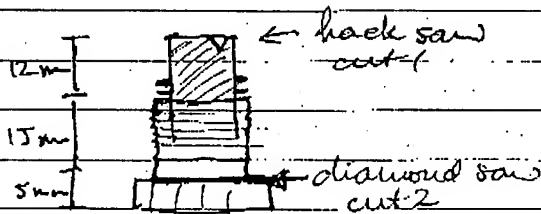
looks infiltrated!



checked under microscope: diamond cut is good enough
to show that the MgB_2 powders are infiltrated
with Mg, forming a Mg/MgB_2 composite.
Took a polaroid, see next page.

▲ open small crucible

• cut 1: Mg to the rim except
small blowhole



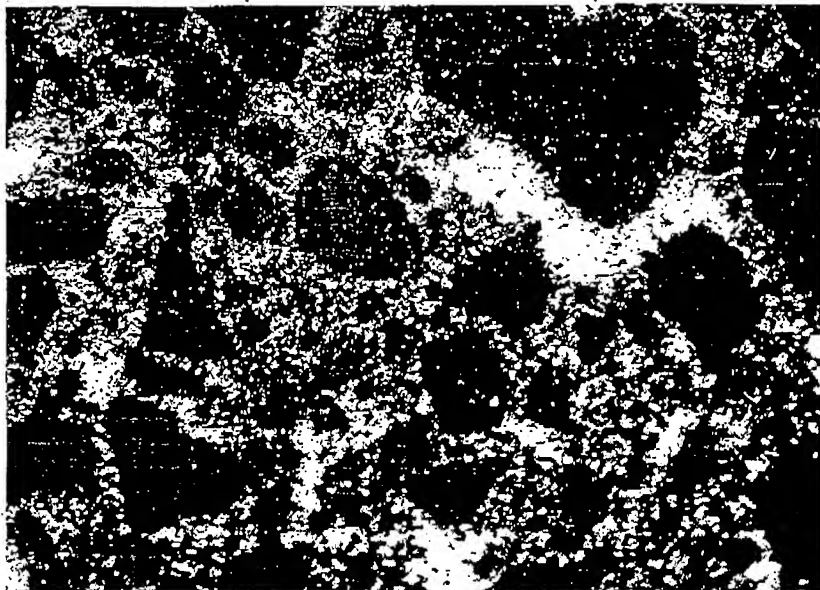
• cut #2 shows only powder, no infiltration

possible problem: 1) leak at nut

2) bad seal by liquid Mg (unlikely)

3) premature solidification (high surface area)

discard sample.



Mg: white
MgB₂: black

$\sim 1/3$ Mg
 $2/3$ MgB₂
should have
continuous
current
path!

Polaroid: plug side of cut 2, very bottom of sample
sample Mg/MgB₂ (1) 40x objective (400x magnification)

- MgB₂ is agglomerated (size $\sim 50-100 \mu\text{m}$)
- Mg infiltrates as vein between agglomerates (veins: $\sim 10-30 \mu\text{m}$)
- many agglomerates are also filled with Mg ($\sim 1-5 \mu\text{m}$)
- some show holes (pull out due to cutting?)

→ this is a fully infiltrated composite, with a few possible regions of porosity (maybe due to closed porosity in original powders or interfering)

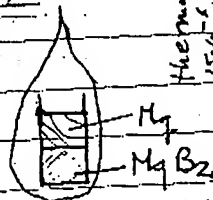
To do: mount & polish this sample

machine steel off

measure conductivity vs T.

next experiments / possible directions)

thermal exp.
15/16" / 100 ± 1.5%
OD: 15.895 mm
DOP: 0.24 mm



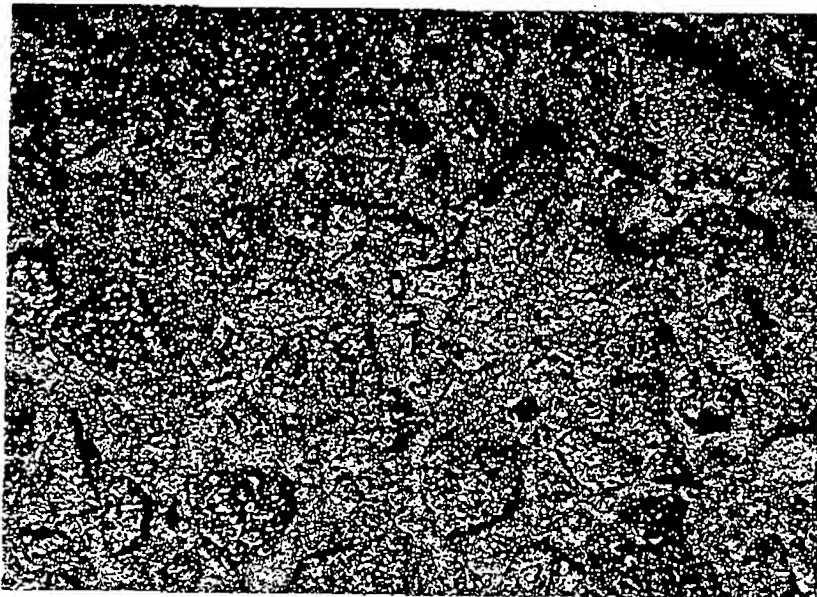
- does Mg wet MgB_2 ?
use steel crucible to encapsulate
 $MgB_2 + Mg$
also may show enhanced sintering due to Mg vapors
(check density of green compact with pycnometer)
- react B fibers in capsule, then infiltrate
- " B powders " " "
- infiltrate B fibers and then react in molten state
- " B powders " " "
- infiltrate MgB_2 with Al } new crucibles
" " " Cu } or BN coated steel

polishing same metallographic section (composite MMC)

1. SiC paper with H_2O
 2. 1 μm diamond in oil (0.05 Al_2O_3 gum up matrix)
- took 2 pictures (see next page)
- composite is successful: 1) full infiltration, no porosity
 - 2) no reaction between Mg & MgB_2
 - 3) no reaction of either phase w. matrix

Machine shop: asked for EDM of MMC
welding of crucibles

Ordered: B powders, MgO powders, more steel



at the edge of composite, saw
pure Mg annulus with some
interdendritic shrinkage porosity.
Not seen in composite

Large (~20-100 μm) Mg B₂
Small (~2-20 μm) Mg B₂



→ need to sieve

Can be added in Mg
matrix

Metals which can be infiltrated in current machine

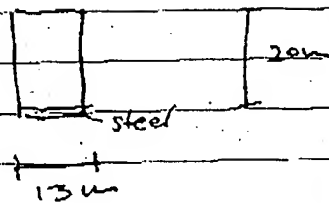
	T_m	T_{ACS}	
Cu	1088 (??)	100%	too hot!!
→ Ag	962	108%	
Al	662	65%	
Mg	650	39%	
Zn	420	28%	
Sn	232	16%	
(Brass 260 953f)		28%	
" 464 900		26%	
ln	156	20%	
pl	327	8%	
Cd	321	25%	
Au	1064	73%	too hot!!

Mutual solubilities in Mg-X systems

	in Mg	in X	intermetallics	eutectic
Mg-Mo	0	0	—	—
Mg-Nb	0	0	—	—
Mg-Co	0	0	MgCo ₂	635
Mg-Cr	0	0	—	no phase diag
Mg-Fe	0	0	—	—
Mg-Be	0	0	1	—
Mg-Nd	0	~2% (sat)	4	528
Mg-Pd	0.23%	2.5at%	7	570
Mg-Pr	0	0	5	575
Mg-Ti	0.12%	1.5at%	—	651
Mg-U	0	6at%	2	483
Mg-Au	0	huge	many	575

mass: 5.213 g

Received machined sample from machine shop. Machinist found composite to be much harder than stainless steel: bolts secure well bonded to matrix.



Side of cylinders show a few pure Hg regions (shiny) but mostly composite

top part was faced → composite (grey)

bottom part is acut from diamond saw

→ need to cut again with diamond saw

The difference of color (grey for machined composite, black for cut composite) is probably due to smearing of Hg during lathe machining.

cut bottom slice (1.5 mm) with diamond saw (or 2h!); other side looks nicely infiltrated, except for a large vein of pure Hg. Unlike the other side, it is not black (could be due to corrosion in). Keep slice and mark it with a "B".
Actually, due to methanol 1 mm in it produces black color it is probably etched by it!

cut upper slice ()

final dimensions:

$$h: 17.044 \text{ mm}$$

$$D: 12.741 \text{ mm}$$

$$m: 4.374 \text{ g}$$

$$r = 6.371 \text{ } \left. \begin{array}{l} h: 17.044 \text{ mm} \\ D: 12.741 \text{ mm} \end{array} \right\} 2.173 \text{ cm}^3$$

$$\rho = 2.011 \text{ g/cc}$$

$$f = 2.63 + (1-f) \cdot 1.74 = 2.011$$

$$f(2.63 - 1.74) = 2.011 - 1.74 = 0.271$$

$$30.5 \text{ vol \% Hg B}_2$$



other sides with

large regions of pure Hg, possibly displaced by falling chunks of Hg upon loading in crucible. In this region, volume factor is larger.

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